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<b>(21) International Application Number:</b> PCT/US97/22493 <b>(22) International Filing Date:</b> 9 December 1997 (09.12.97)  <b>(30) Priority Data:</b> 08/869,876                      5 June 1997 (05.06.97)                      US  <b>(71) Applicant:</b> EXXON CHEMICAL PATENTS INC. [US/US]; 5200 Bayway Drive, Baytown, TX 77520-5200 (US).  <b>(72) Inventors:</b> DHARMARAJAN, Narayanaswami, Raja; 14406 Fair Knoll Way, Houston, TX 77062 (US). RAVIS- HANKAR, Periagaram, Srinivasan; 6006 Soaring Pine Court, Kingwood, TX 77345 (US). BURRAGE, Charles, Douglas; 20003 Pinehurst Place Drive, Humble, TX 77346 (US).  <b>(74) Agents:</b> MILLER, Douglas, W.; Exxon Chemical Company, P.O. Box 2149, Baytown, TX 77522-2149 (US) et al.		<b>(81) Designated States:</b> AU, BR, CA, CN, JP, KR, MX, RU, European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report.</i>
<b>(54) Title:</b> ELECTRICAL DEVICES INCLUDING ETHYLENE, ALPHA-OLEFIN, VINYL NORBORNENE ELASTOMERS AND ETHYLENE ALPHA-OLEFIN POLYMERS  <b>(57) Abstract</b>  Elastomeric polymers including ethylene, alpha-olefin and vinyl norbornene are shown to have improved extrusion characteristics, improved electrical properties, improved cure characteristics compared to ethylene, alpha-olefin, non-conjugated diene elastomeric polymers containing non-conjugated dienes other than vinyl norbornene. The elastomeric polymers containing vinyl norbornene generally have a branching index below 0.5. Such elastomeric polymers are combined with up to 30 parts of metallocene catalyzed ethylene, alpha-olefin copolymers to improve physical properties.		

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**ELECTRICAL DEVICES INCLUDING ETHYLENE, ALPHA-OLEFIN,  
VINYL NORBORNENE ELASTOMERS AND ETHYLENE ALPHA-  
OLEFIN POLYMERS**

5     **TECHNICAL FIELD**

      This invention relates to electrically conductive or semi-conductive devices having an insulating member including an ethylene,  $\alpha$ -olefin, vinyl norbornene elastomeric polymer having a branching index of less than 0.5 and a metallocene catalyzed ethylene  $\alpha$ -olefin copolymer and the compounds made from the elastomeric polymer ethylene-copolymer combination providing elastomeric polymer based members having excellent surface characteristics and dielectric strength.

10     **BACKGROUND**

      Typical power cables generally include one or more conductors in a core that is generally surrounded by several layers that can include a first polymeric semi-conducting shield layer, a polymeric insulating layer and a second polymeric semi-conducting shield layer, a metallic tape and a polymeric jacket. A wide variety of polymeric materials have been utilized as electrical insulating and semi-conducting shield materials for power cable and numerous other electrical applications.

20     In elastomer or elastomer-like polymers often used as one or more of the polymer (insulation) members in power cables, common ethylene,  $\alpha$ -olefin, non-conjugated diene elastic polymers materials that have come into wide use usually include ethylene,  $\alpha$ -olefin, and a non-conjugated diene selected from the group consisting of 5-ethylidene-2-norbornene (ENB), 1,4-hexadiene (HD), 1,6 octadiene, 5-methyl-1,4 hexadiene, 3,7-dimethyl-1,6-octadiene, and the like. Such polymers can provide a good insulating property for power cables. However, generally selection of one of these elastomeric polymers while bringing certain advantages, also brings some disadvantages as well. For instance, electrical compounds containing some of these polymers usually necessitate slower extrusion rates than might be desirable for optimum output, because surface characteristics of the extrudate in a compound based on these elastomeric polymers will not be as smooth as desired if the extrusion rates are higher. By choosing elastomeric

polymers containing 5-vinyl-2-norbornene (VNB), a higher level of extrusion output can be achieved in the compound usually due to a higher level of branching, but some diminution of physical properties (over the less branched material) may result.

5 Much of the production of insulated electrical devices would see an advantage from higher throughput or extrusion rates (eg, lowering manufacturing costs) but many of the so called conventional elastomeric polymers, especially those based on 1, 4-Hexadiene and ENB, would exhibit a tendency to cure slowly.

10 There is a commercial need for an elastomeric polymer ethylene alpha-olefin copolymer blend insulating material for electrical devices that can be extruded relatively rapidly, in the substantial absence of surface roughness, having a relatively rapid cure rate, relatively high cure state and relatively low electrical loss. There is also a need for improved long term heat aging and lower cure additives consumption, all of which may reduce the overall manufacturing cost and/or improve quality of the cable insulation.

#### SUMMARY

20 We have discovered that polymeric insulation for electrical conducting devices, when it includes an ethylene, alpha-olefin, vinyl norbornene elastomeric polymer with a relatively low branching index, indicative of long chain branching, will provide a smooth surface at relatively high extruder speeds, and generally will cure faster to a higher cure state than previously available ethylene, alpha-olefin, non-conjugated diene elastomeric polymers. Additionally, inclusion of a minority component of metallocene catalyzed ethylene alpha-olefin copolymer (hereinafter m-ethylene copolymer) enhances the physical properties of the elastomeric polymer in an electrical insulating compound based on the combination or blend.

25 According to one embodiment of our invention, an electrically conductive device is provided including (a) an electrically conductive member comprising at least one electrically conductive substrate; and (b) at least one electrically insulating member in proximity to the electrically conductive member. In this embodiment the insulating member includes an elastomeric polymer selected from 30 the group consisting of ethylene, polymerized with at least one  $\alpha$ -olefin, and vinyl norbornene. The insulating member also includes up to 30, preferably up to 25,

more preferably up to 20, most preferably up to 15 parts per hundred parts of elastomeric polymer (pphep) of a m-ethylene copolymer.

5 The elastomeric polymers of various embodiments of our invention may contain in the range of from 50-90 mole percent ethylene preferably 65-90 mole percent, more preferably 68-80 mole percent based on the total moles of the polymer. The elastomeric polymer contains the alpha-olefin in the range of from 10-50 mole percent, preferably in the range of from 10-35 mole percent, more preferably in the range of from 20-32 mole percent. The elastomeric polymers will have a vinyl norbornene content in the range of from 0.16-5 mole percent, more preferably 0.16-1.5 mole percent, most preferably 0.16-0.4 mole percent based on 10 the total moles of the polymer. The elastomeric polymer will also have a Mooney viscosity (ML [ 1+4] 125 °C ) generally in the range of from 10-80. The elastomeric polymer will have a  $M_{w, GPC, LALLS} / M_{n, GPC, DRI}$  ( $M_w/M_n$ ) greater than 6.

15 The m-ethylene copolymer will generally be a copolymer of ethylene and at least one alpha-olefin, selected from one of butene-1, 4-methyl-1-pentene, pentene-1, hexene-1, octene-1 and combinations thereof. Such copolymers may be generally characterized in that they will have a  $M_w/M_n$  of 3 or less, and a Composition Distribution Breadth Index (CDBI) greater than 50%. Electrical 20 insulating and/or semi-conducting compounds using these elastomeric polymer blends with m-ethylene copolymers, may be made using fillers and other constituents well known to those of ordinary skill in the art.

To attain the same cure state as commercially available ethylene, alpha-olefin, non-conjugated diene elastomeric polymers with the diene selected for 25 example from the group consisting of 5-ethylidene-2-norbornene, 1,4-hexadiene, 1,6 octadiene, 5-methyl-1,4 hexadiene, 3,7-dimethyl-1,6-octadiene, and the like, the elastomeric polymer/m-ethylene copolymer blends described in an embodiment of our invention require lower diene levels, at substantially equivalent curative levels.

30 Alternatively, at the same diene content as these other ethylene, alpha-olefin, non-conjugated diene elastomeric polymers, lower curative levels will be necessary to reach the same or a higher cure state. The ethylene, alpha-olefin,

vinyl norbornene elastomeric polymers of certain embodiments of our invention have a branching index below 0.5. The lower branching index representative of the majority component of the blend permits the extruded insulating members to have a smoother surface at higher extrusion rates and a lower die swell compared to previously available commercial materials. Owing to lower diene content, the ethylene, alpha-olefin, vinyl norbornene elastomeric polymers of certain embodiments of our invention, required to achieve the same cure state as previously available ethylene, alpha-olefin, non-conjugated diene elastomeric polymer, the compounds formulated with the elastomeric polymers of our invention generally exhibit improved heat aging performance relative to the previously available ethylene, alpha-olefin, non-conjugated diene elastomeric polymer compounds. This heat aging benefit also accrues to the blends of the present invention.

These and other features, aspects, and advantages of the present invention will become better understood with reference to the following description and appended claims.

#### **BRIEF DESCRIPTION OF DRAWINGS**

These and other features, aspects, and advantages of the present invention will become better understood with reference with the following description, appended claims, and accompanying drawings where:

**Figure 1** shows the variation of tensile strength of a compound based on the blend with the percent of m-ethylene copolymer as part of the compound.

**Figure 2** shows the variation of elongation of a compound based on the blend with percent of m-ethylene copolymer as a part of the compound.

**Figure 3** shows the variation of cure rate properties of a compound based on the blend with percent m-ethylene copolymer level.

**Figure 4** shows the variation of cure state of a compound based on the blend with percent m-ethylene copolymer level.

**Figure 5** shows the variation of mass rate with extruder speed for both blended and unblended elastomeric polymers.

**Figure 6** shows the variation of extrudate roughness with screw speed for both blended and unblended elastomeric polymers.

Figure 7 shows the variation of extrudate roughness with mass rate for both blended and unblended elastomeric polymers.

All compounds described in figures 1-7 are based on a formulation called Superohm 3728 as shown in Table 9, containing 60 parts of clay.

Figure 8 shows the compositional distribution of elastomeric polymers made with different co-catalysts.

## **DESCRIPTION**

### **Introduction**

Various embodiments of the present invention concern certain elastomeric polymer m-ethylene copolymer blend compositions, certain compound compositions and applications based on the elastomeric polymer and the compounds made therefrom. These elastomeric polymer m-ethylene copolymer blend compositions have properties when used in an electrically conducting device which make them particularly well-suited for applications that require excellent surface characteristics, faster cure rates, more complete cure state, lower amounts of curative agent, and improved dielectric properties.

Following is a detailed description of various preferred elastomeric polymer m-ethylene copolymer blend compositions within the scope of the present invention, preferred methods of producing these compositions, preferred m-ethylene copolymers that may be included in the compound, and preferred applications of these polymer compositions. Those skilled in that art will appreciate that numerous modifications of these preferred embodiments can be made without departing from the scope of this invention. For example, although the properties of the elastomeric polymer and m-ethylene copolymer blend composition and compounds based on the blend are exemplified in electrical insulating applications, they will have numerous other electrical uses. To the extent our description is specific, it is solely for purpose of illustrating preferred embodiments of our invention and should not be taken as limiting the present invention to these specific embodiments.

The use of headings in the present application is intended to aid the reader, and is not intended to be limiting in any way.



Various values given in the text and claims are determined and defined as follows.

No.	Test	Test Method	Units
1	Branching Index	Exxon (described here)	none
2	(elastomeric polymer composition determination) Ethylene	ASTM D 3900	wt%
3	Ethylidene Norbornene Vinyl Norbornene	FT. - Infra Red FT. - Infra Red	wt% wt%
4	Mooney Viscosity	ASTM D 1646 - 94	Mooney Units
5	Scorch Time	ASTM D 2084 - 93	minutes
6	Cure Characteristics $M_L$ $M_H$ $t_{s2}$ $t_{c90}$ Cure State = ( $M_H - M_L$ ) Cure Rate	ASTM D 2084 - 93	dN.m dN.m minutes minutes dN.m dN.m / min
7	100 % Modulus	ASTM D 412 - 92	MPa
8	300 % Modulus	ASTM D 412 - 92	MPa
9	Tensile Strength	ASTM D 412 - 92	MPa
10	Elongation	ASTM D 412 - 92	%
11	Surface Roughness (R)	Surfcom® 110B Surface gauge	$\mu\text{m}$
12	Extrusion  Mass Rate Screw Speed	Haake Rheocord 90 Extruder. Die Temperature = 110-125°C. Screw Speed = 25-95 RPM. Extruder L/D = 16/1. Die Diameter = 3.2 mm, Die Land Length = 9.5 mm, Die (L/D) = 3/1, Compression Screw = 2/1, Garvy Die and Round hole Die.	  g / min rpm

Various physical properties of compounds based on the elastomeric polymer, m-ethylene copolymer blends of certain embodiments of our invention and ranges for these properties are shown below. By compound we intend that the common elastomer industry meaning of the word be employed. That is the polymer component(s) (in the present application a blend) in combination with fillers, accelerants, curatives, extenders and the like are what make up a compound. All properties ascribed to compounds based on the blends of embodiments of the



present invention are determined based on a compound (Superohm 3728) as shown in Table 9, unless otherwise specified. It should be understood that by changing amounts and proportions of such a compound physical properties can be effected. Such property differences can be seen for example in Tables 5-8, based on 30 phr clay. For this reason the subsequently claimed properties for compounds utilizing the blends are intended as a reference to the performance of the blends and should not be limited to the specific compound. Various physical properties of compounds based on the elastomeric polymers of certain embodiments of our invention and ranges of these properties are shown below.

	Test Condition	Units	Broad	Preferred	More Preferred
I	Compound Properties				
	ML (1+8) 100°C	MU	< 35	< 33	< 30
	Cure State (MH-ML))	dN.m	> 90	> 92	
	Cure Rate	dN.m/min	>100	>110	>112
	Tensile Strength	MPa	> 8.5	> 9	> 10
	Elongation	%	> 250	> 275	> 300
II	Extrusion Properties @ 125°C				
	Surface Roughness ( $R_t$ ) @ 55 RPM	$\mu\text{m}$	< 50	< 45	
	Mass Extrusion Rate @ 55 RPM	g/min	> 65	> 70	> 75

In general peroxide levels in such compounds may be described as follows:

	Test Condition	Units	Broad	Narrow	Very Narrow
	Peroxide Level				
	Dicumyl Peroxide	(gm mole/phr) $\times 10^{-3}$	3 to 89	3 to 45	9 to 25

In certain embodiments of the present invention, an electrically conductive device comprises: a) an electrically conductive member including at least one electrically conductive substrate; and b) at least one electrically insulating member substantially surrounding the electrically conducting member including a polymer being one of ethylene polymerized with an  $\alpha$ -olefin and a non-conjugated diene, said  $\alpha$ -olefin is one of propylene, butene-1, 4-methyl-1-pentene, hexene-1, octene-

1, decene-1, or combinations thereof, said non-conjugated diene being vinyl norbornene, wherein said polymer has a branching index (BI) (defined below) of up to 0.5, preferably the BI of the elastomeric polymer is up to 0.4, more preferably up to 0.3, most preferably up to 0.2; and a m-ethylene copolymer having a CDBI 50%, preferably 55%, more preferably 60%, most preferably 65%, and a  $M_w/M_n$  less than 3, preferably less than 2.5. The insulating member includes up to 30, preferably up to 25, more preferably up to 20, most preferably up to 15 parts per hundred parts of elastomeric polymer (pphep) of a m-ethylene copolymer also expressed as 5-30 pphep, 5-25 pphep, 5-20 pphep and 5-15 pphep.

**The Ethylene, Alpha-Olefin, Vinyl Norbornene Elastomeric Polymer**

The Ziegler polymerization of the pendent double bond in vinyl norbornene (VNB) is believed to produce a highly branched ethylene, alpha-olefin, vinyl norbornene elastomeric polymer. This method of branching permits the production of ethylene, alpha-olefin, vinyl norbornene elastomeric polymers substantially free of gel which would normally be associated with cationically branched ethylene, alpha-olefin, vinyl norbornene elastomeric polymer containing, for instance, a non-conjugated diene such as 5-ethylidene-2-norbornene, 1,4-hexadiene, and the like. The synthesis of substantially gel-free ethylene, alpha-olefin, vinyl norbornene elastomeric polymers containing vinyl norbornene is discussed in Japanese laid open patent applications JP 151758, JP 210169, which we incorporated by reference herein for purposes of U.S. patent practice.

Preferred embodiments of the aforementioned documents to synthesize polymers suitable for this invention are described below:

The catalyst used are  $VOCl_3$  (vanadium oxytrichloride) and  $VCl_4$  (vanadium tetrachloride) with the latter as the preferred catalyst. The co-catalyst is chosen from (i) ethyl aluminum sesqui chloride (SESQUI), (ii) diethyl aluminum chloride (DEAC) and (iii) equivalent mixture of diethyl aluminum chloride and triethyl aluminum (TEAL). As shown in Figure 8, the choice of co-catalyst influences the compositional distribution in the polymer. The polymer with broader compositional distribution is expected to provide the best tensile strength in the dielectric cable compound. The polymerization is carried out in a continuous stirred tank reactor at 20-65° C at a residence time of 6-15 minutes at a pressure of

7 kg/cm<sup>2</sup>. The concentration of vanadium to alkyl is from 1 to 4 to 1 to 8. About 0.3 to 1.5 kg of polymer is produced per gm of catalyst fed to the reactor. The polymer concentration in the hexane solvent is in the range of 3-7% by weight. The synthesis of ethylene, alpha-olefin, vinyl norbornene polymers were conducted both in a laboratory pilot unit (output 4 Kg/day), a large scale semi works unit (output 1T/day), and a commercial scale production unit (output 200,000 kg/day).

A discussion of catalysts suitable for polymerizing our elastomeric polymer or other catalysts and co-catalysts contemplated are discussed in the two Japanese laid open patent applications incorporated by reference above.

The resulting polymers had the following molecular characteristics:

The intrinsic viscosity measured in decalin at 135° C were in the range of 1 - 2 dl/g. The molecular weight distribution ( $M_{w,LALLS}/M_{n,GPC/DRI}$ ) was >10. The branching index was in the range 0.1 - 0.3.

Metallocene catalysis of the above monomers is also contemplated including a compound capable of activating the Group 4 transition metal compound of the invention to an active catalyst state is used in the invention process to prepare the activated catalyst. Suitable activators include the ionizing noncoordinating anion precursor and alumoxane activating compounds, both well known and described in the field of metallocene catalysis.

Additionally, an active, ionic catalyst composition comprising a cation of the Group 4 transition metal compound of the invention and a noncoordinating anion result upon reaction of the Group 4 transition metal compound with the ionizing noncoordinating anion precursor. The activation reaction is suitable whether the anion precursor ionizes the metallocene, typically by abstraction of R<sub>1</sub> or R<sub>2</sub>, by any methods inclusive of protonation, ammonium or carbonium salt ionization, metal cation ionization or Lewis acid ionization. The critical feature of this activation is cationization of the Group 4 transition metal compound and its ionic stabilization by a resulting compatible, noncoordinating, or weakly coordinating (included in the term noncoordinating), anion capable of displacement by the copolymerizable monomers of the invention. See, for example, EP-A-0 277,003, EP-A-0 277,004, U.S. Patent No. 5,198,401, U.S. Patent No. 5,241,025, U.S. Patent No. 5,387,568, WO 91/09882, WO 92/00333, WO 93/11172 and WO

94/03506 which address the use of noncoordinating anion precursors with Group 4 transition metal catalyst compounds, their use in polymerization processes and means of supporting them to prepare heterogeneous catalysts. Activation by alumoxane compounds, typically, alkyl alumoxanes, is less well defined as to its mechanism but is none-the-less well known for use with Group 4 transition metal compound catalysts, see for example U.S. Patent No. 5,096,867. Each of these documents are incorporated by reference for purposes of U.S. patent practice.

For peroxide cure applications, vinyl norbornene containing ethylene, alpha-olefin, diene monomer elastomeric polymers require lower levels of peroxide to attain the same cure state compared to ethylene, alpha-olefin, diene monomer with ethylidene norbornene termonomer at the same level of incorporated diene. Typically 20 to 40 % lower peroxide consumption can be realized using ethylene, alpha-olefin, vinyl norbornene. The efficiency of vinyl norbornene in providing high cross link density with peroxide vulcanization also permits a reduction in the overall diene level to attain the same cure state as ethylidene norbornene polymers. This results in enhanced heat aging performance, generally owing to lower diene incorporation. This unique combinations of improved processability, lower peroxide usage and enhanced heat aging are the benefits provided by ethylene, alpha-olefin, vinyl norbornene over conventional non-conjugated dienes such as ethylidene norbornene or 1-4, hexadiene or the like including terpolymer or tetrapolymers.

The relative degree of branching in ethylene, alpha-olefin, diene monomer is determined using a branching index factor. Calculating this factor requires a series of three laboratory measurements<sup>1</sup> of polymer properties in solutions. These are: (i) weight average molecular weight ( $M_{w,LALLS}$ ) measured using a low angle laser light scattering (LALLS) technique; (ii) weight average molecular weight ( $M_{w,DRI}$ ) and viscosity average molecular weight ( $M_{v,DRI}$ ) using a differential refractive index detector (DRI) and (iii) intrinsic viscosity (IV) measured in decalin at 135° C. The first two measurements are obtained in a GPC using a filtered dilute solution of the polymer in tri-chloro benzene.

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<sup>11</sup>VerStrate, Gary "Ethylene-Propylene Elastomers", Encyclopedia of Polymer Science and Engineering, 6, 2nd edition, (1986)

An average branching index is defined as:

$$BI = \frac{M_{v,br} \times M_{w,DRI}}{M_{w,LALLS} \times M_{v,DRI}} \quad (1)$$

5 where,  $M_{v,br} = k(IV)^{1/a}$ ;

and 'a' is the Mark-Houwink constant (= 0.759 for ethylene, alpha-olefin, diene monomer in decalin at 135° C).

From equation (1) it follows that the branching index for a linear polymer is 1.0, and for branched polymers the extent of branching is defined relative to the linear polymer. Since at a constant  $M_n$ ,  $(Mw)_{branch} > (Mw)_{linear}$ , BI for a branched polymers is less than 1.0, and a smaller BI value denotes a higher level of branching. It should be noted that this method indicates only the relative degree of branching and not a quantified amount of branching as would be determined using a direct measurement, i.e. NMR.

#### 15 Ethylene Alpha-Olefin Copolymer Component

The ethylene alpha-olefin copolymers that are produced using metallocene catalysts include ionizing activators as well as alumoxanes is included as a component of the elastomeric polymer compound component can be any polyethylene, as long as the polyethylene component is a polyethylene with the following features:

	<u>preferred</u>	<u>more preferred</u>	<u>most preferred</u>
$M_w/M_n$	<3	<2.5	
CDBI	>50%	>60%	>65%
$M_z/M_n$	<2		

Generally these ranges dictate the use of a m-ethylene copolymer with a density in the range of from 0.86-0.920, preferred 0.865-0.915, more preferred 0.87-0.91 g/cc, most preferred 0.88-0.90 g/cc. Densities referred to herein will generally be polymer or resin densities, unless otherwise specified.

25 There is a wide variety of commercial and experimental m-ethylene copolymer resins useful in the manufacture of electrical insulation included in certain embodiments of the present invention. A non-inclusive list is found below along with the general bulk resin properties as published:

TABLE A

Commercial Designation	Density (g/cm <sup>3</sup> )	Melt Index (g/10 min.)	Type
Exact® 3017*	0.901	27	eth/butene
Exact® 3022*	0.905	9.0	eth/butene
Exact® 4006*	0.880	10	eth/butene
Exact® 4033*	0.880	0.8	eth/butene
Exact® 4041*	0.878	3.0	eth/butene
Engage® 8100**	0.870	1.0	eth/octene

\* available from Exxon Chemical Co., Houston, TX, USA

\*\* available from Dow Chemical Co., Freeport, TX, USA

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It will be understood that in general we contemplate that a large number of m-ethylene copolymers will be useful in the techniques and applications described herein. Included components: ethylene propylene copolymers, ethylene-1-butene copolymers, ethylene-1-hexene copolymers, ethylene-1-octene copolymers, ethylene-4-methyl-1-pentene copolymers, ethylene 1-decene copolymers, ethylene-1-pentene copolymers, as well as ethylene copolymers of one or more C4 to C20 containing alpha-olefins, and combinations thereof. A nonexclusive list of such polymers: ethylene, 1-butene, 1-pentene; ethylene, 1-butene, 1-hexene; ethylene, 1-butene, 1-octene; ethylene, 1-butene, decene; ethylene, 1-pentene, 1-hexene; ethylene, 1-pentene, 1-octene; ethylene, 1-pentene, decene; ethylene, 1-octene; 1-pentene; ethylene 1-octene, decene; ethylene, 4-methyl-1-pentene, 1-butene; ethylene 4-methyl-1-pentene, 1-pentene; ethylene, 4-methyl-1-pentene, 1-hexene; ethylene 4-methyl-1-pentene, 1-octene; ethylene, 4-methyl-1-pentene, decene. Included in the m-ethylene copolymers will be one or more of the above monomers included at a total level of 0.2-30 mole percent, preferably 0.5-4 mole percent, or such mole percents consistent with the resin densities contemplated.

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Definitions and methods of determinations of CDBI for substantially crystalline polymers may be found in U.S. 5,008,204 which is fully incorporated by reference herein for purposes of US patent practice. For polymers that are substantially amorphous, determination of CDBI is somewhat different. Techniques for such polymers may be found in U.S. 5,191,042 (specifically for instance at column 10, lines 47-68) which is fully incorporated by reference herein



for purposes of US patent practice. Additionally, in Ver Strate, G., "Structure Characterization in the Science and Technology of Elastomers", Academic Press, Inc., *Science And Technology Of Rubber*, 1978, pp. 75-144, which is fully incorporated by reference herein for purposes of US patent practice.

5       The resin and product properties recited in this specification were determined in accordance with the following test procedures. Where any of these properties is referenced in the appended claims, it is to be measured in accordance with the specified test procedure.

Property	Units	Procedure
Melt Index	dg/min.	ASTM D-1238(E)
Density	g/cc	ASTM D-1505

10       Preferred melt indexes for the m-ethylene copolymer are 0.5-150 preferably 0.5-50, more preferably 0.5-30 dg/min.

#### EXAMPLES

15       Ethylene, alpha-olefin, vinyl norbornene polymers are synthesized at diene levels varying from 0.3 to 2 weight percent and evaluated in medium voltage electrical compound formulations. A major portion of the compound data and replicate measurements are obtained with ethylene, alpha-olefin, vinyl norbornene elastomeric polymer having a diene content of 0.8 weight percent. Little benefit is observed in increasing the diene level beyond 1 weight percent, as it is possible to reduce the diene level below 1% and still retain both a high state of cure and substantial levels of branching. Table 1 shows the polymer characteristics of  
20       several ethylene, alpha-olefin, non-conjugated diene elastomeric polymers. Polymer 3 (comparative) is a commercially available ethylene, propylene, 1,4-hexadiene elastomeric polymer, Nordel® 2722 (available from E.I. DuPont). The ethylene, alpha-olefin, vinyl norbornene polymer from the commercial plant is referenced as Polymer 7. Table 1 shows the polymer characteristics of all the  
25       elastomeric polymers used in the compound formulations. Polymer 7 has a higher level of branching compared to polymer 3. The branching index for Polymer 7 is 0.1, while for Polymer 3 BI is > 0.5.

#### Compound Extrusion Characterization



Extrusion studies of the electrical compounds are performed in a Haake Rheocord® 90 extruder ( $L/D = 16/1$ ). A screw with a compression ratio of 2/1 (geometry typical for processing rubber compounds) is used in all extrusions. The extrusion temperature is maintained in the range of 110 to 125°C. The extruder screw speed is varied from 30 to 90 rpm so that extrusion properties could be monitored at varying extrusion rates. Samples are obtained after the torque and the pressure drop equilibrated to a steady value at a constant screw speed.

The mass throughput and the surface roughness of the extrudate are measured at different extruder screw speeds. The mass throughput is represented as the weight of the extrudate per unit time.

The surface roughness of the extrudate is measured using a Surfcom® 110 B surface gauge (manufactured by Tokyo Seimitsu Company). The Surfcom® instrument contains a diamond stylus which moves across the surface of the sample subject to evaluation. This sample can range in hardness from metal or plastic to rubber compounds. The instrument records the surface irregularities over the length (assessment length) traveled by the diamond stylus. This surface roughness is quantified using a combination of two factors:

1.  $R_a$  ( $\mu\text{m}$ ), an arithmetic mean representing the departure of the extrudate surface profile from a mean line.
2.  $R_t$  ( $\mu\text{m}$ ), the vertical distance between the highest point and the lowest point of the extrudate roughness profile within the assessment length.

The Roughness Factor (R) is defined as:

$$R (\mu\text{m}) = R_a + 0.1 R_t .$$

and incorporates both the  $R_a$  and  $R_t$  terms.  $R_t$  is given a lower weighting to adjust for its magnitude relative to  $R_a$ . In some cases the extrudate roughness is qualified by just  $R_t$ , the vertical distance between the highest and lowest point.

**Blends of ethylene, alpha-olefin, vinyl norbornene elastomeric polymer with m-ethylene copolymer**

Table 2 shows medium voltage electrical compound formulations containing 30 phr clay with other additives. Herein phr (parts per hundred rubber)

is used interchangeably with pphep. The clay, Translink® 37, is a calcined surface modified (vinyl modification) Kaolin available from Engelhard. Table 3 shows m-ethylene copolymers used in the examples. All of the compounding is performed in a 1600 cc Banbury internal mixer. The mixing conditions and procedures are shown in Table 4. The compounds discharged from the Banbury mixer were sheeted out in a two roll mill. The peroxide was added in the mill to 300 grams of the compound.

Table 5 compares the cure characteristics and physical properties of Polymer 7, (Example 1), Polymer 7 with Exact® 4033 (Example 2) and Exact® 4033 as the sole polymer (Example 3). The peroxide level is maintained at 6.5 phr in all the formulations. The peroxide used in the compounds of Table 5 is Dicap 40 KE, which is a 40 % active dicumyl peroxide supported on Burgess clay. The cure rate in Example 1 with the VNB containing elastomeric polymer is significantly higher than Example 1 and Example 3 compounds. All three examples (1, 2, and 3) have generally the same cure state. The tensile strength and elongation of Examples 2 and 3 are high compared to Example 1.

Table 6 shows the extrusion characteristics of Examples 1, 2, and 3. The mass extrusion rate increases with increasing extruder screw speed, but the extrudate surface generally worsens. At any given extruder speed, increasing the concentration of m-ethylene polymer in the compound enhances throughput but coarsens the extrudate surface. The m-ethylene copolymer compound with m-ethylene as the sole polymer (Example 3) shows significant melt fracture at low shear rates. The surface roughness of the compound containing the blend of VNB elastomeric polymer with m-ethylene copolymer (Example 2), even though coarser than Example 1, is still within acceptable limits.

Table 7 shows the cure characteristics of the compounds of Examples 4-8. The m-ethylene polymers listed in Table 3 were used in formulating with Polymer 7. Except for a small decrease in cure rate, addition of m-ethylene polymer does not alter cure characteristics relative to the control compound (Example 4). In the same examples, the addition of the m-ethylene copolymer improves the compounds tensile strength by 19% and elongation by 20%. Table 8 shows the extrusion characteristics of the same examples (4-8). The surface (characteristics of

Examples 5 through 8 containing the m-ethylene copolymer) is somewhat rougher than Example 4, but is generally unacceptable from a surface roughness standpoint. The mass throughput of these compounds is higher than that of control Example 4.

An additional sub-set of these examples other m-ethylene copolymer (Exact® 3017) of lower molecular weight or higher melt index, were compounded with the elastomeric polymer and other ingredients. Table 9 shows the formulations with 60 phr clay, cure characteristics and physical properties of these compounds. Table 10 shows the extrusion characteristics of these compounds (Examples 9-13), showing that at a constant shear rate the mass throughput is enhanced over the compound of unblended elastomeric polymer. Table 9 shows, that the cured characteristics of these compounds again leads to improvements in physical properties (primarily elongation) over the non-blended compound. Figures 1 and 2 show the effect of adding Exact® 3017 (m-ethylene copolymer) on compound tensile strength and elongation. There is a decrease in tensile strength, which levels off, but elongation increases continuously. For comparison the performance of the compound containing Polymer 3 (Nordel® 2722) is also shown in the figures. At around 20 phr Exact® 3017 concentration, both tensile strength and elongation exceed the performance of Polymer 3 formulation. Figures 3 and 4 illustrate the effect of Exact® 3017 concentration on the cure rate and cure state respectively. The cure rate declines gradually with increasing m-ethylene copolymer, while the cure state passes through a maximum. The cure state in all the compositions is significantly higher than Polymer 3 (Nordel® 2722) formulation, while the cure state matches the Polymer 3 compound at around 15 phr Exact® 3017 concentration.

Figure 5 shows the variation in mass extrusion rate with extruder screw speed for formulation with the VNB elastomeric polymer (Polymer 7), Nordel® 2722 (Polymer 3), as well as blends of the VNB elastomeric polymer with 10 and 20 phr of Exact® 3017. All compounds with the VNB elastomeric polymer, and blends of Polymer 7 with Exact® 3017 have a higher mass throughput at all extrusion speeds compared to the Nordel® 2722 (Polymer 3) formulation.

Figure 6 shows the variation in surface roughness, ( $R_t$ ), with extrusion speed in a 60 phr clay formulation. A lower  $R_t$  value indicates a smoother surface.

The comparative formulation with Polymer 3 (Nordel® 2722) shows significant melt fracture at 75 RPM which further deteriorates to a very rough surface at an extruder screw speed of 95 RPM. The formulation where the elastomeric VNB polymer (Polymer 7) is the sole component has the best extrusion surface. The blended formulation of Polymer 7 with Exact® 3017 have surface characteristics coarser than Polymer 7 compound, but significantly improved over the Polymer 3 formulation.

Figure 7 shows the variation of surface roughness with extruder throughput (mass rate) for the various formulations. The formulation with Nordel® 2722 (Polymer 3) transitions vary rapidly to significant melt fracture at higher throughputs. As expected the elastomeric VNB polymer formulation (Polymer 7) shows the smoothest extrudate. The blended compounds with m-ethylene copolymer have extrudate appearance that is significantly smoother than the control Polymer 3 formulation, at high mass rates.

Although the present invention has been described in considerable detail with reference to certain preferred embodiments thereof, other versions are possible. For example, other elastomeric polymers and m-ethylene copolymers and other uses also contemplated. Additionally, while certain ingredients have been exemplified, other ingredients, and/or other inclusion levels are also contemplated. Therefore the spirit and scope of the appended claims should not be limited to the description of the preferred versions contained herein.

**TABLE 1**  
**POLYMER CHARACTERISTICS**

POLYMER	ML (1+4) 125 °C	Ethylene (wt. %)	Diene Type	Diene (wt. %)	BI *
Polymer 3 <sup>(a)</sup>	23	74	HEX <sup>(1)</sup>	4.0	0.6
Polymer 7 <sup>(b)</sup>	31	76	VNB <sup>(2)</sup>	0.87	0.10

\* Branching Index

(1) 1,4-Hexadiene

(2) 5-Vinyl-2-norbornene

(a) E.I. duPont, Nordel® 2722

(b) Exxon Chemical Company

**TABLE 2**  
**MEDIUM VOLTAGE ELECTRICAL COMPOUNDS WITH 30 PHR CLAY**

Components	Description	Formulation (phr)
Polymer <sup>1</sup>		100
Translink 37	Calcined Clay	30
Agerite MA	Antioxidant	1.5
Drimix A 172	Vinyl Silane	1.0
Zinc Oxide		5.0
ERD 90	Red Lead	5.0
Escorene LD 400	Low Density Polyethylene	5.0
Paraffin 1236	Wax	5.0
Curatives Dicup 40 KE	Dicumyl peroxide on clay (40% Active)	6.5

<sup>1</sup> Includes Polymer 7 and blends of Polymer 7 with Exact® and Engage® polymers

**TABLE 3**  
**ETHYLENE ALPHA-OLEFIN COPOLYMER POLYMER**  
**CHARACTERISTICS**

POLYMER	Comonomer	Melt Index (dg/min)	Density (g/cc)	ML*	Ethylene (wt. %)	Diene Type	Diene (wt. %)
Exact® <sup>3</sup> 4033	C4	0.8	0.880	28	75	none	0
Exact® 4006	C4	10	0.880		75	none	0
Exact® 4041	C4	3	0.878	10		none	0
Exact® 3022	C4	9	0.905			none	0
Engage® <sup>4</sup> 8100	C8	1	0.870	20	76	none	0

<sup>3</sup> Exxon Chemical Company

<sup>4</sup> Dow Chemical

\* ML @ (1+4) 125°C

**TABLE 4**  
**TYPICAL MIXING PROCEDURE**

Equipment : 1600 cc. Banbury Mixer

Batch Size : 1100 gm

Time (minutes)	Rotor Speed (RPM)	Ingredients Addition
0	85	Polymer, Agerite
0.5	85	1/2 Clay, Zinc Oxide, ERD 90, 1/2 Drimix, LD 400
2.0	100	1/4 Clay, 1/4 Drimix, 1/2 Wax
3.0	100	1/4 Clay, 1/4 Drimix, 1/2 Wax
4.0	100	Sweep
5.5	100	Sweep
7.0		Dump

**TABLE 5**  
**CURE CHARACTERISTICS AND PHYSICAL PROPERTIES OF**  
**COMPOUNDS**  
**CONTAINING POLYMER 7 WITH EXACT ® 4033**  
**Compound formulation per Table 2**

Example		1	2	3
Polymer 7	phr	100	85	-
Exact® 4033	phr	-	15	100
Mooney Scorch (MS) - 132 °C, min. to 3 point rise	min	17.8	14.2	12.9
Compound Mooney Viscosity (ML) 100 °C (1+8) minutes	Mooney Units	33	44	219
<u>Rheometer 200 °C, 6 min motor, 3° Arc</u>				
M <sub>L</sub>	dN.m	7.2	9.8	10.5
M <sub>H</sub>	dN.m	93.1	94.8	93.6
t <sub>s2</sub>	min	0.59	0.68	0.57
t <sub>c90</sub>	min	1.76	1.96	1.96
Cure Rate	dN.m/min	113.2	99.1	75.8
M <sub>H</sub> -M <sub>L</sub>	dN.m	85.9	85.0	83.1
<u>Cure 20 min., 165 °C</u>				
Hardness	shore A	84	84	91
100 % Modulus	MPa	4.3	3.8	5.8
200 % Modulus	MPa	6.7	6.2	7.9
300 % Modulus	MPa	-	9.0	10.1
Tensile Strength	MPa	8.3	9.9	15.1
Elongation	%	265	319	522

**TABLE 6**  
**EXTRUSION CHARACTERISTICS OF COMPOUNDS**  
**CONTAINING POLYMER 7 WITH EXACT ® 4033**  
**Compound formulation per Table 2**

Example		Screw Speed (rpm)	1	2	3
Polymer 7	phr		100	85	-
Exact® 4033	phr		-	15	100
Mass Extrusion Rate	g/min	30	40.9	44.9	49.4
Extrudate Roughness - R Factor	µm		6.2	3.6	2.6
Mass Extrusion Rate	g/min	60	82.0	84.3	97.0
Extrudate Roughness - R Factor	µm		4.3	8.3	23.7
Mass Extrusion Rate	g/min	90	114.9	110.0	140.4
Extrudate Roughness - R Factor	µm		3.8	9.1	too rough

**TABLE 7**  
**CURE CHARACTERISTICS AND PHYSICAL PROPERTIES OF**  
**COMPOUNDS CONTAINING POLYMER 7 WITH EXACT ® AND**  
**ENGAGE ® POLYMERS**

Compound formulation per Table 2

Example		4	5	6	7	8
Polymer 7	phr	100	85	85	85	85
Exact® 4006	phr	-	15	-	-	-
Exact® 4041	phr	-	-	15	-	-
Exact® 3022	phr	-	-	-	15	-
Engage® 8100	phr	-	-	-	-	15
Mooney Scorch (MS) - 132 °C, time to 3 point rise	min	17.8	17.9	15.1	16.6	14.7
Compound Mooney Viscosity (ML) 100 °C (1+8) minutes	Mooney Units	33	31	36	34	37
<u>Rheometer 200 °C, 6 min motor, 3° Arc</u>						
M <sub>L</sub>	dN.m	7.2	6.6	7.7	6.9	8.2
M <sub>H</sub>	dN.m	93.1	90.3	94.6	94.6	92.2
t <sub>s2</sub>	min	0.59	0.63	0.58	0.62	0.60
t <sub>c90</sub>	min	1.76	1.87	1.81	1.86	1.83
Cure Rate	dN.m/min	113.2	100.3	103.8	105.1	101.3
M <sub>H</sub> -M <sub>L</sub>	dN.m	85.9	83.7	86.9	87.7	84.0
<u>Cure 20 min., 165 °C</u>						
Hardness	shore A	84	85	85	86	84
100 % Modulus	MPa	4.3	4.3	3.8	4.9	3.6
200 % Modulus	MPa	6.7	7.0	6.4	7.6	6.2
Tensile Strength	MPa	8.3	9.4	9.4	10.2	8.8
Elongation	%	265	286	293	299	292

**TABLE 8**  
**EXTRUSION CHARACTERISTICS OF FORMULATIONS CONTAINING**  
**POLYMER 7 WITH EXACT ® AND ENGAGE ® POLYMERS**  
 Compound formulation per Table 2

Example		Screw Speed (rpm)	4	5	6	7	8
Polymer 7	phr		100	85	85	85	85
Exact® 4006	phr		-	15	-	-	-
Exact® 4041	phr		-	-	15	-	-
Exact® 3022	phr		-	-	-	15	-
Engage® 8100	phr		-	-	-	-	15
Mass Extrusion Rate	g/min	30	40.9	45.7	47.9	46.8	41.5
Extrudate Roughness - R Factor	µm		6.2	8.4	11.5	4.2	9.0
Mass Extrusion Rate	g/min	60	82.0	85.5	87.5	84.2	84.2
Extrudate Roughness - R Factor	µm		4.3	5.2	6.0	4.7	8.3
Mass Extrusion Rate	g/min	90	114.9	108.6	125.4	120.5	114.8
Extrudate Roughness - R Factor	µm		3.8	3.5	6.4	5.6	8.5



**TABLE 9**  
**CURE CHARACTERISTICS AND PHYSICAL PROPERTIES OF**  
**COMPOUNDS CONTAINING EXACT® 3017 POLYMER WITH**  
**60 PHR CLAY**

5

Compound No.		9	10	11	12	13
<b>INGREDIENT</b>	<b>PHR</b>					
Polymer 3		100	-	-	-	-
Polymer 7		-	100	95	90	80
EXACT ® 3017		-	-	5	10	20
Translink 37 Clay		60	60	60	60	60
Agerite MA		1.5	1.5	1.5	1.5	1.5
Drimix A 172		1	1	1	1	1
Zinc Oxide		5	5	5	5	5
ERD 90		5	5	5	5	5
Escorene LD 400		5	5	-	-	-
Paraffin Wax 1236		5	5	5	5	5
DiCup 40 KE		6.5	6.5	6.5	6.5	6.5
Specific Gravity	1.2					
Total PHR	189.0					
Batch Factor	6.35					
Batch Weight (gm)	1200					
Fill Factor (%)	63					
<b>COMPOUND MOONEY VISCOSITY</b>						
ML@ (1+8) 100 °C	MU	38	31	29	29	25
<b>MOONEY SCORCH</b>						
Ms(t3), 132° C	min	20	11	12	20	21
<b>ODR 200 °C, 3° arc</b>						
ML, minimum	dN.m	9	7	7	6	5
MH, maximum	dN.m	108	103	107	104	98
ts2, scorch	min	0.7	0.5	0.6	0.6	0.6
tc90, cure	min	2.0	1.7	1.7	1.8	1.8
Cure Rate	dN.m/min	97	134	138	129	115
MH-ML, cure state	dN.m	94	96	100	98	93
<b>PHYSICAL PROPERTIES</b>						
press cured , 20 min @ 165 °C						
Hardness	shore A	89	87	85	89	88
100 % Modulus	MPa	5.6	6.7	5.7	5.7	5.9
200 % Modulus	MPa	8.7	10.1	8.9	8.7	8.7
300 % Modulus	MPa	x	x	x	x	10.0
Tensile Strength	MPa	10.0	11.0	9.9	10.0	10.4
Elongation At Break	%	295	240	260	275	330

**TABLE 10**  
**EXTRUSION OF ELECTRICAL COMPOUNDS**  
**CONTAINING EXACT® 3017 POLYMER**

5

Extruder : HAAKE Rheocord 90  
 2:1 CR Screw  
 Die Diameter : 0.125 inches  
 Die Land Length : 0.375 inches  
 Die Temperature : 125° C

Cmpd. No.	Polymer	RPM	Shear Rate (1/sec)	Mass Rate (g/min)	Vol. Rate (cc/min)	Die Pressure Drop (MPa)	Surface Roughness, R <sub>t</sub> (μm)
9	Polymer 3	25	130	31	25	6.0	37
		35	170	40	32	6.6	18
		55	255	60	49	7.5	54
		75	370	86	70	8.3	69
		95	450	105	85	8.6	too rough
10	Polymer 7	25	145	34	27	5.9	36
		35	220	52	42	6.7	29
		55	330	77	62	7.6	26
		75	445	103	84	8.2	32
		95	540	126	102	8.5	26
12	EXACT® 3017 (10 phr)	25	155	36	29	5.7	21
		35	210	48	39	6.3	25
		55	340	78	64	7.3	40
		75	460	107	87	8.0	57
		95	515	120	97	8.3	50
13	EXACT® 3017 (20 phr)	25	150	35	28	5.4	28
		35	215	50	40	6.1	32
		55	350	81	66	7.1	53
		75	450	104	85	7.7	44
		95	545	126	102	8.2	48

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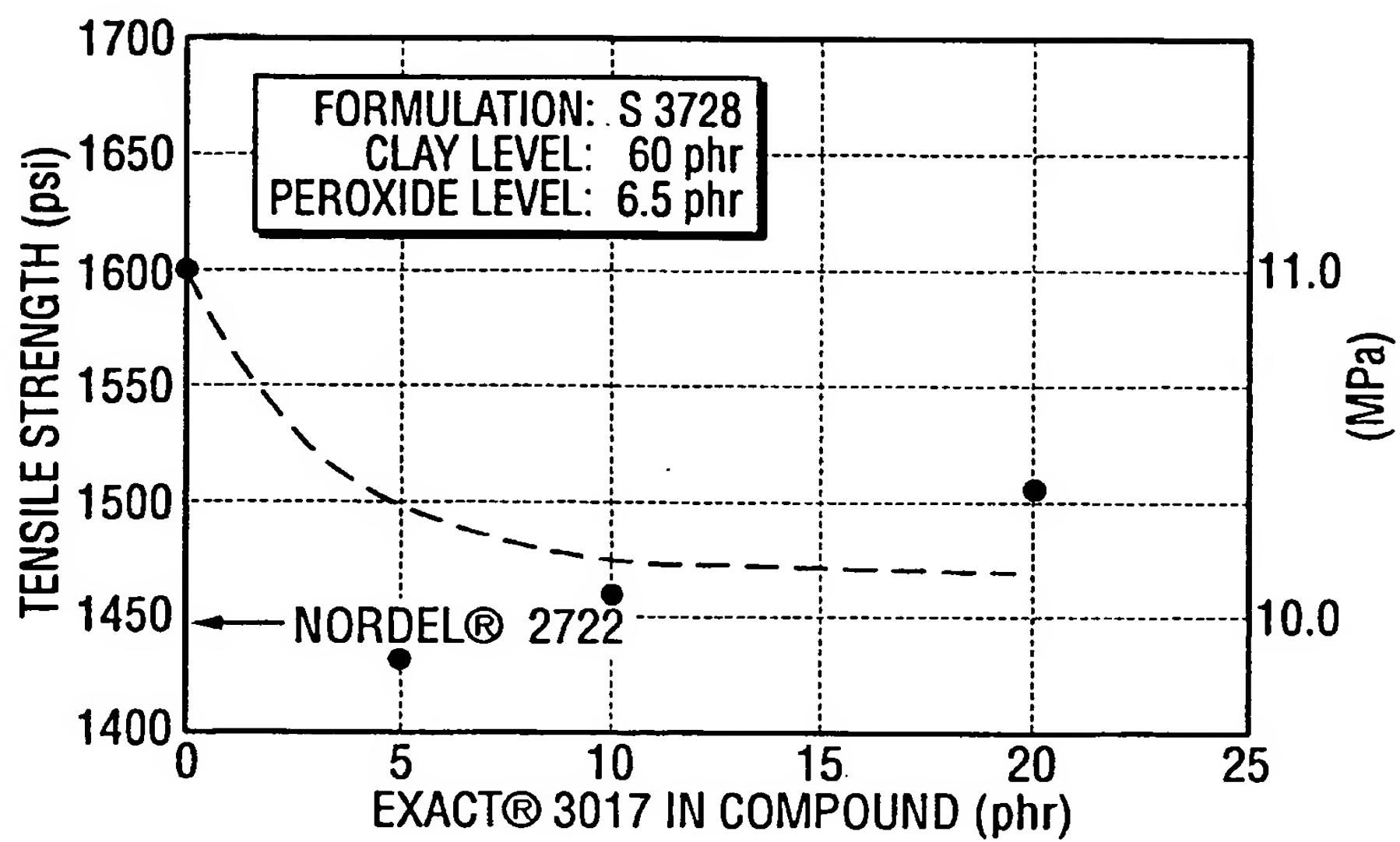
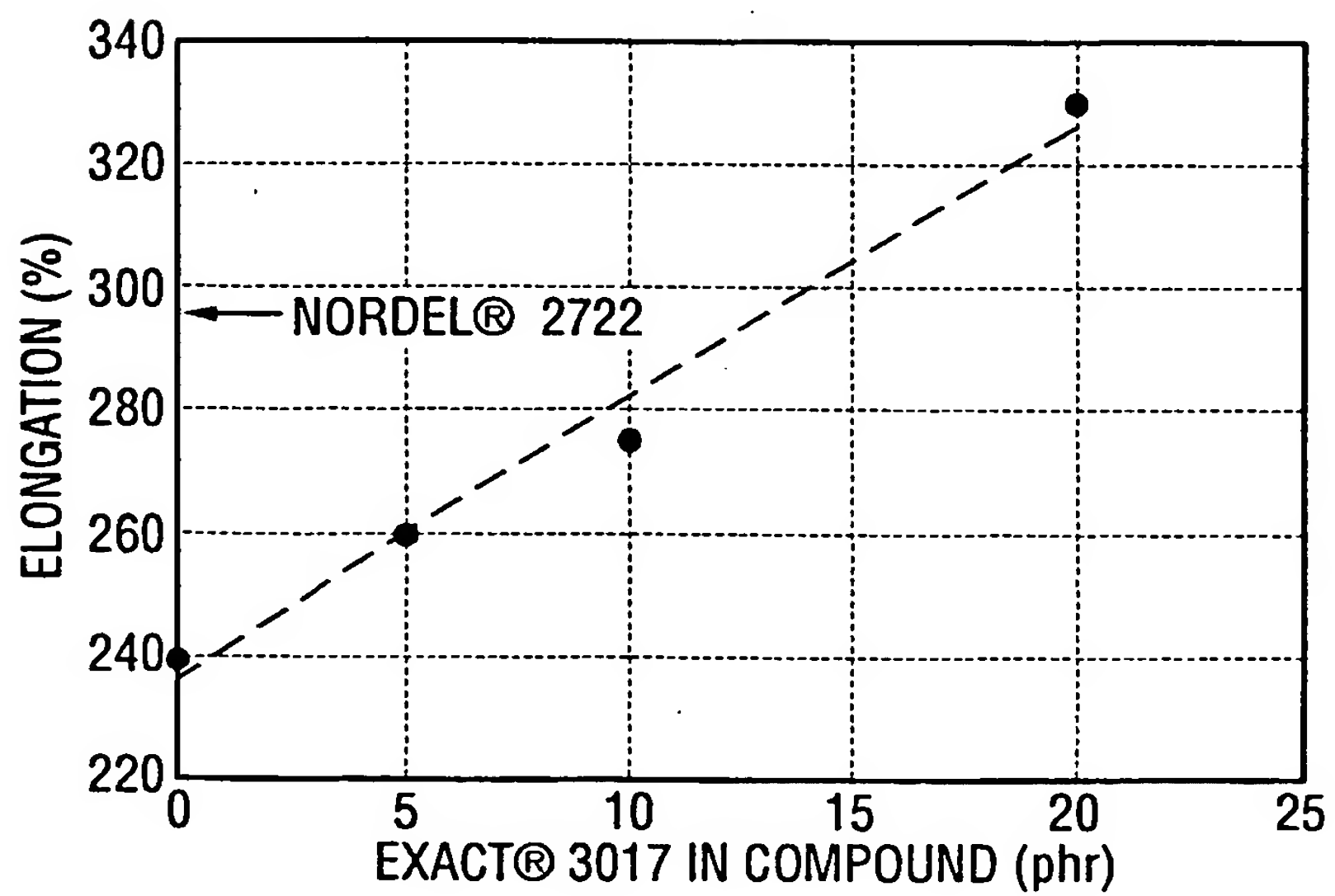
Based on formulation of Table 9

**CLAIMS****We Claim:**

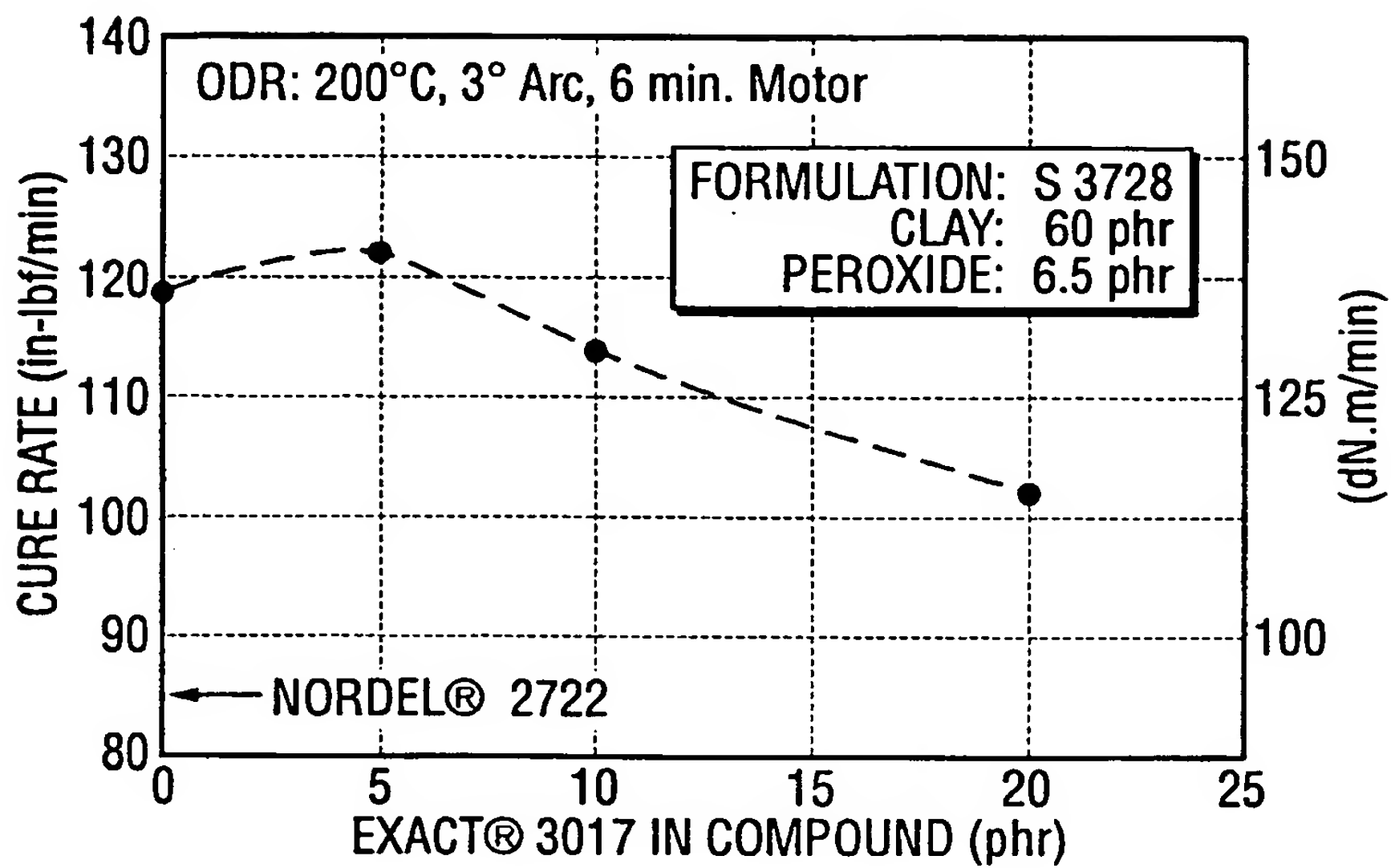
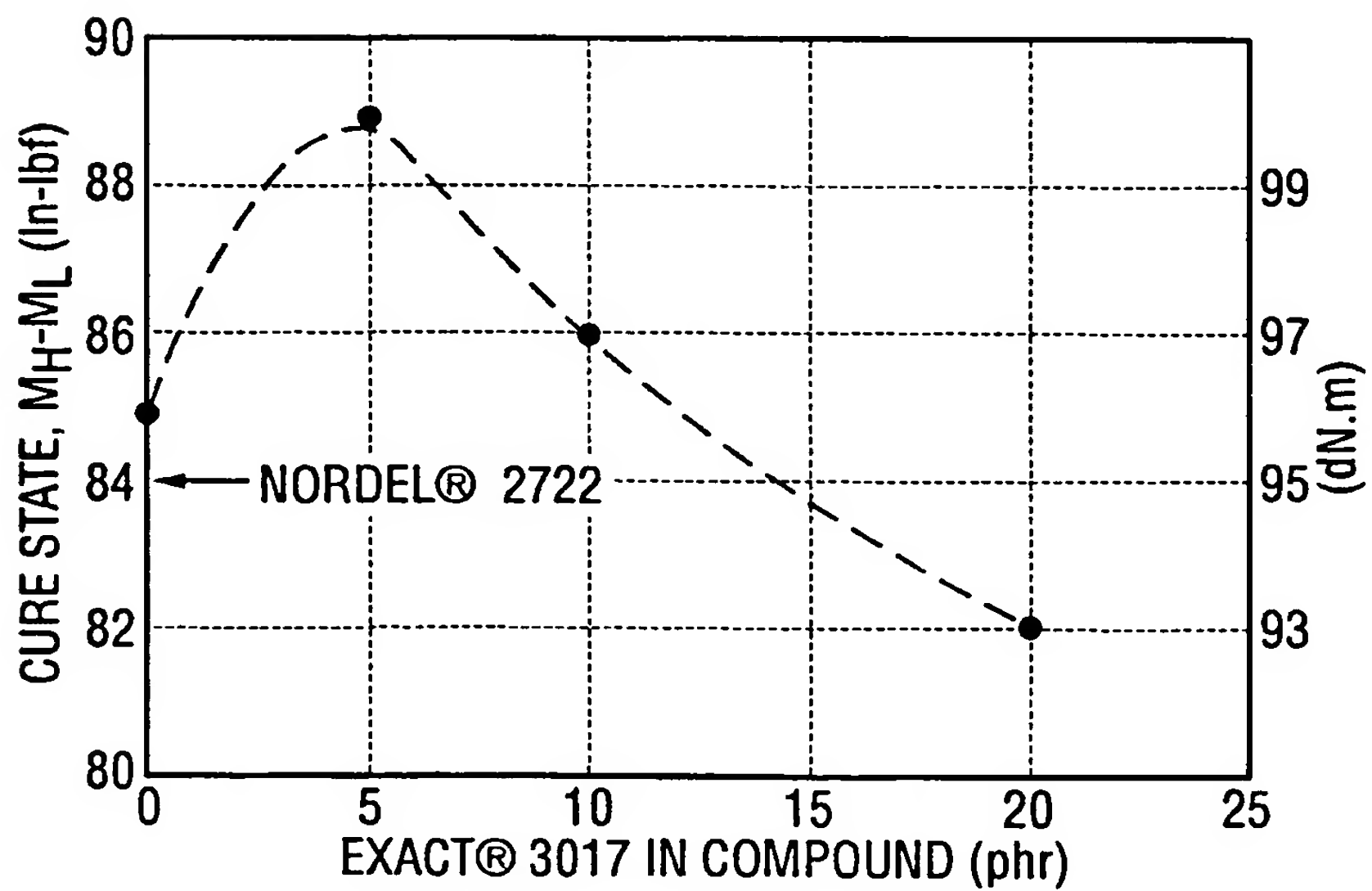
- 5 1. A cable coating compound based on Superohm 3728, having an electrical dissipation factor less than 0.6%, a Mooney viscosity of less than 35 ML (1+8) 100° C, a cure state above 90 dN.m/min, a tensile strength above 8.5 MPa, a cure rate above 100 dN.m/min, and an elongation above 250%, comprising:
  - 10 a) an ethylene, propylene, 5-vinyl-2-norbornene elastomeric polymer having a branching index up to 0.4, and a  $M_w/M_n$  greater than 10; and
  - b) 5-20 parts per hundred parts of said elastomeric polymer of an ethylene alpha-olefin copolymer having a  $M_w/M_n$  less than 3, a CDBI greater than 50% and a density in the range of from 0.86-0.92 g/cc.
- 15 2. The cable coating compound of claim 1 having an electrical dissipation factor less than 0.5%, wherein said compound has an ML (1+8) 100° C less than 33, a cure rate above 112 dN.m/min, a tensile strength above 10 MPa, and an elongation above 300%, wherein:
  - 20 a) said elastomeric polymer, has a branching index below 0.02 and a  $M_w/M_n$  greater than 10;
  - b) said ethylene alpha-olefin copolymer has an  $M_w/M_n > 2.5$ , a CDBI > 60%, preferably >65% and a density in the range of from 0.865-0.915 g/cc; and preferably from 0.87-0.91 g/cc.
- 25 3. A power cable coating compound comprising:
  - a) an ethylene, alpha-olefin, vinyl norbornene elastomeric polymer and
  - b) an ethylene alpha-olefin copolymer said copolymer having a  $M_w/M_n$  less than 3.
  - 30 c) said elastomeric polymer has a branching index less than 0.4, an ethylene content in the range of 50-90 mole percent, a vinyl norbornene content in the range of 0.16-1.5 mole percent, and a  $M_w/M_n$  greater than 10; and
  - d) said ethylene alpha-olefin copolymer has a CDBI greater than 60% wherein a compound including said elastomeric polymer and said ethylene alpha-olefin copolymer has a cure rate above 100 dN.m/min, a cure state above 90 dN.m/min, a tensile strength above 9 MPa and an elongation above 250%.
- 35

4. The cable coating compound of any of the preceding claims wherein said compound is cross-linked with at least one of cross-linking agents or radiation, preferably wherein said cross-linking agent is dicumyl peroxide.
5. Use of the cable coating compound of any of the preceding claims to coat an electrically conductive member.
6. The cable coating compound of claim 5 wherein said conductive member is selected from the group consisting of aluminum, copper and steel.

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**FIG. 1****FIG. 2**

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**FIG. 3****FIG. 4**

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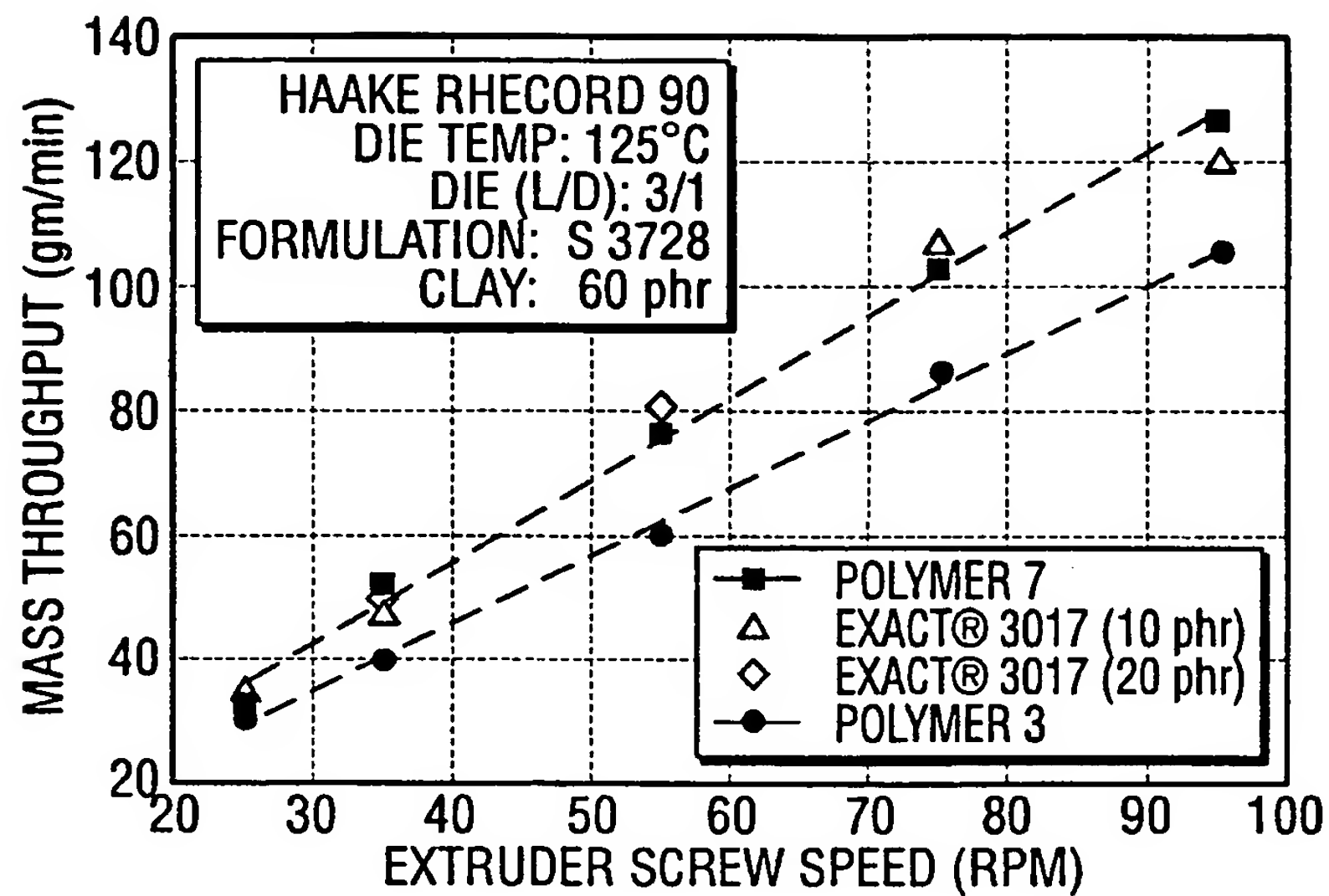
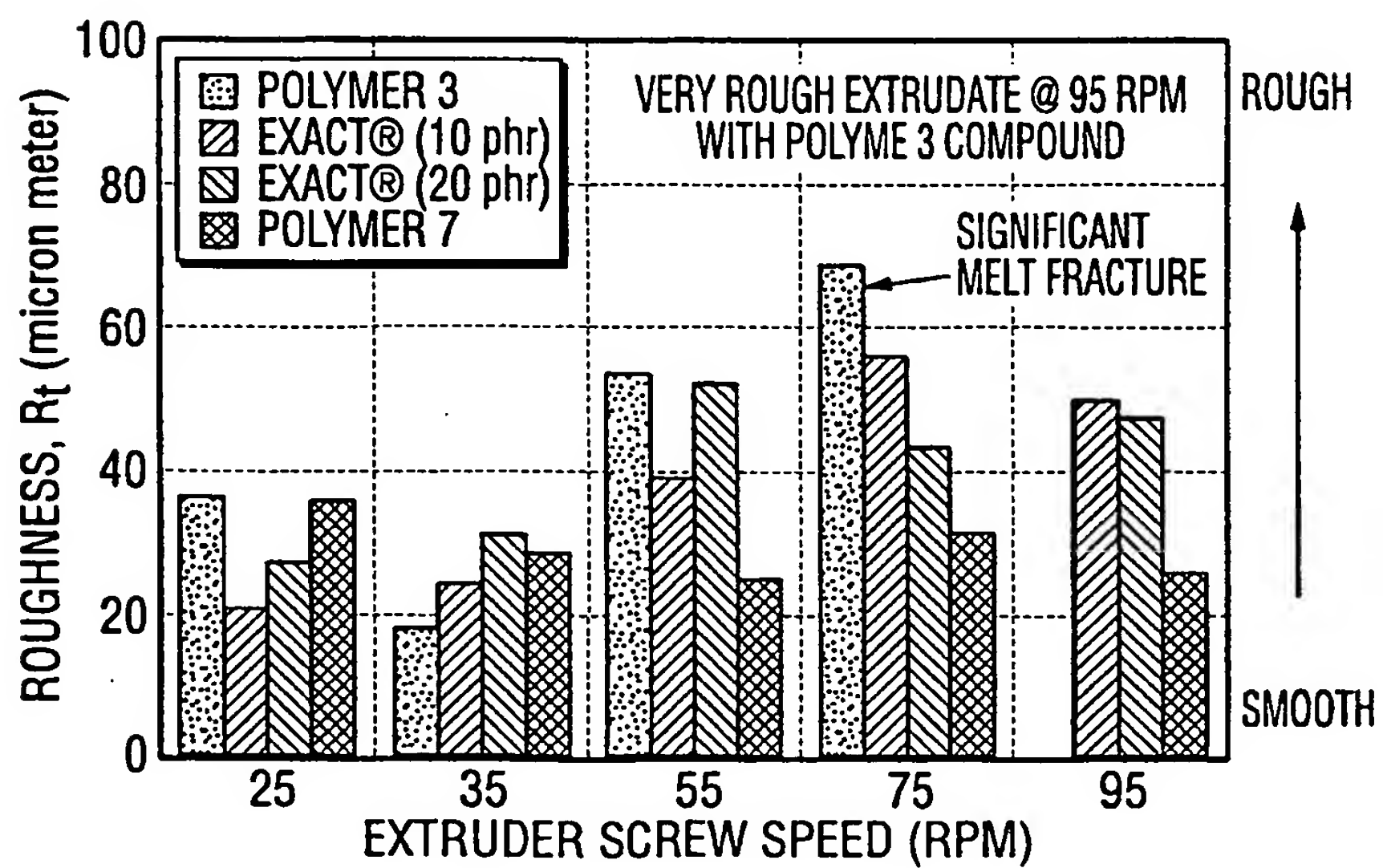
**FIG. 5****FIG. 6**



FIG. 7

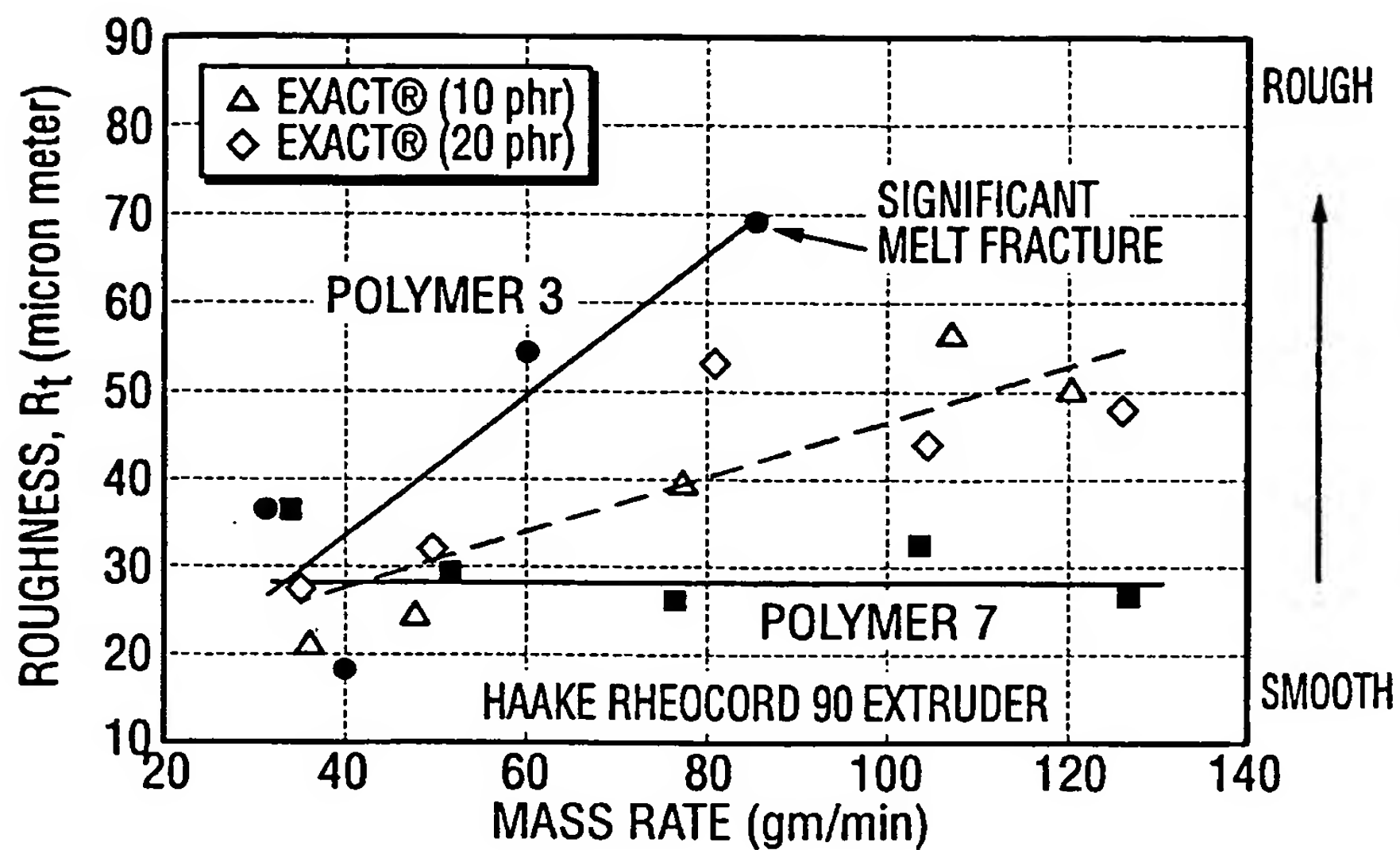
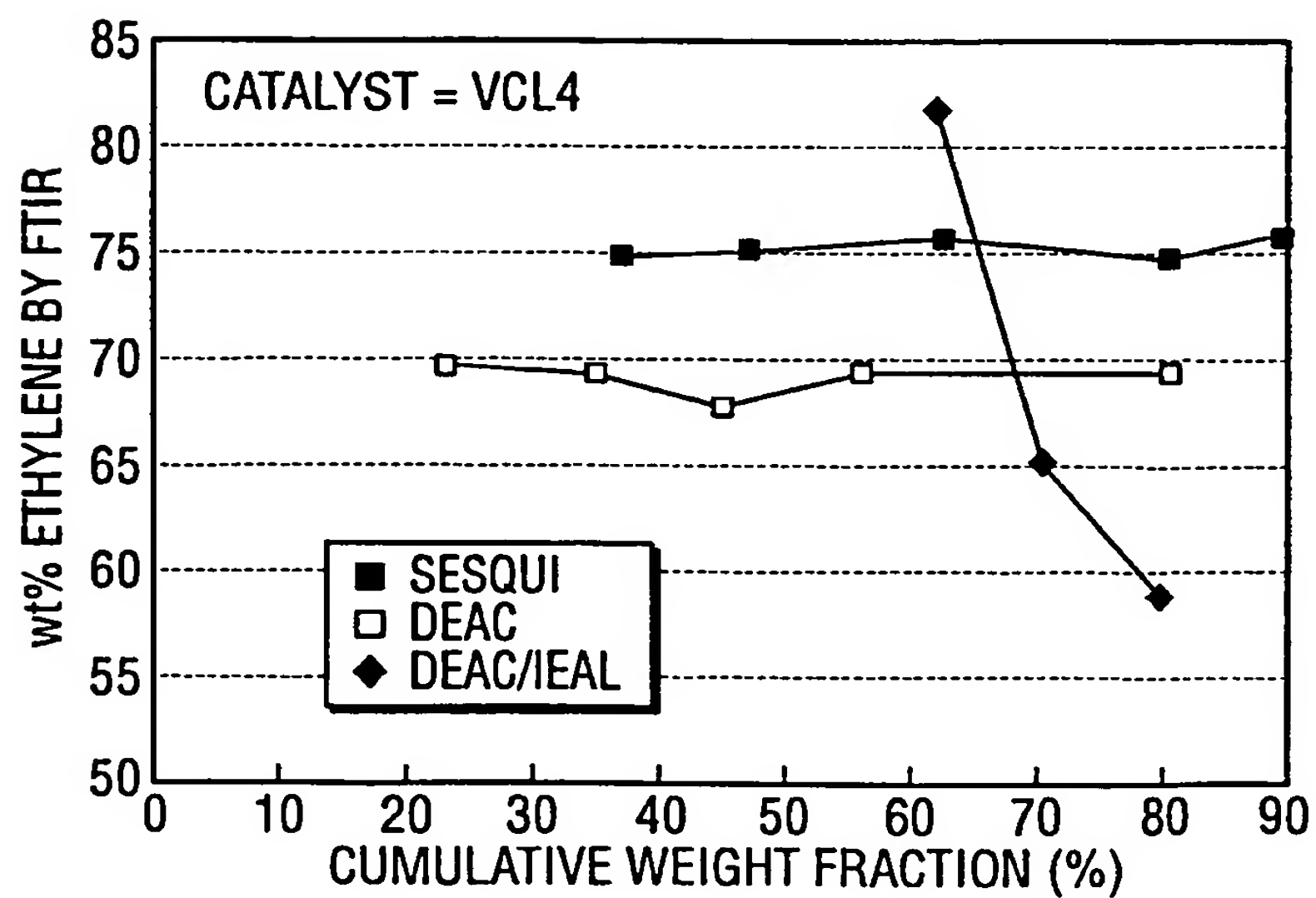


FIG. 8



# INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/22493

A. CLASSIFICATION OF SUBJECT MATTER  
IPC 6 H01B3/44

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 H01B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 97 00523 A (EXXON CHEMICAL PATENTS INC) 3 January 1997	
A	EP 0 005 320 A (EXXON RESEARCH ENGINEERING CO) 14 November 1979	
A	US 5 246 783 A (SPENADEL LAWRENCE ET AL) 21 September 1993	

☐ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

### \* Special categories of cited documents :

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Date of the actual completion of the international search

29 April 1998

Date of mailing of the international search report

11/05/1998

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# INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 97/22493

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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